ADA

DISTILLATE FUEL TRENDS: INTERNATIONAL SUPPLY VARIATIONS AND ALTERNATE FUEL PROPERTIES

INTERIM REPORT
TFLRF No. 435

by
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Southwest Research Institute® (SwRI®)
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for
Patsy Muzzell
U.S. Army TARDEC
Force Projection Technologies
Warren, Michigan

Contract No. W56HZV-09-C-0100 (WD04–Tasks II-VI & Tasks IX-XI)

January 2013

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14. ABSTRACT

The U.S. Army uses two fuels in the majority of its vehicles, aircraft, and support equipment. They are diesel fuel and jet fuel. These two fuels, and the specifications that govern them around the world, have undergone significant changes over the past two decades. There is a general trend toward a more uniform diesel around the world but the use of alternative fuels, such as biodiesel, has introduced additional variations in the world market. Aviation fuel, by comparison, is moving from a fairly narrowly defined, semi-formal set of standards to a formal international requirement. This report contains a discussion of the recent changes, as well as possible future changes, in the specifications and composition of these two important fuels.

In addition to a comparison of specifications and composition, a series of inspection tests was performed on selected alternate diesel and jet fuels. Although there were some failures to meet specification limits, it was remarkable how often an alternate fuel that was clearly not suitable for routine use, passed the conformance testing. This work demonstrated that conformance to a specification is often insufficient as proof of fit for purpose conformance.

15. SUBJECT TERMS

Alternative Fuel, Aviation Fuel, Kerosene, Diesel Fuel, Jet Fuel

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EXECUTIVE SUMMARY

The primary distillate fuels, diesel fuel, and jet fuel, are moving in different directions. While there are international goals to make a more uniform diesel fuel product, the reality is the market is becoming more diverse, not less. Aviation turbine fuel, by comparison is moving from fairly narrow defined semi-formal set of standards to a formal international requirement.

The intent of this program was to gather some unique samples from domestic and international sources. This was accomplished with limited success. While there was a lot of interest in providing alternative fuels, there was a lot less actual product being generated. Still, a sufficient number of samples were obtained to provide a glimpse at the opportunities and problems that are associated with alternative fuels.

Diesel fuel is local market driven. Even in standards that cover multiple regions and countries, like ASTM D975 and EN 590, there are numerous versions of the standard to balance cost and performance issues. While as an overall trend diesel fuel has become a better product, driven primarily by environmental efforts to reduce sulfur; however, there are still areas with high sulfur, poor stability fuel.

The primary source of alternate diesel fuel is Biodiesel, more properly FAME (Fatty Acid Methyl Ester). Depending on the percentage used, its inclusion may or may not be noted at the point of sale, but most diesel fuel in NATO countries will have some amount of FAME present. There is some work being done on hydrocarbon alternatives but the regulatory structure favors the emphasis on FAME.

While diesel fuel has a wide variety of types and specifications, kerosene jet fuel is close to an ideal commodity. Essentially, there is only one kerosene jet fuel in the world. True, there is a difference in freeze point between Jet A and Jet A1 but except for a very few flights that is a moot point. The fact is, for all the properties that define the day to day performance of jet fuel, the requirements are uniform worldwide.

All the efforts in alternative jet fuel are aimed at enforcing the same uniformity of product. The aviation industry has developed an approval program that ensures any new product is “fit for purpose” as a jet fuel. The main effort is to develop fuel pathway process specific formulation information that allows the alternative material to be used as a jet fuel component without limitation to the final product. The final product is then considered identical to the refined products.

Part of this program was to run a series of inspection tests on the alternate diesel and jet fuels received. There were some failures, as might be expected, but it was more remarkable how often something that clearly is not suitable as diesel or jet fuel passed the conformance tests. The key point to understand about specification testing is that it is only as reliable as the quality and integrity of the sample. If the material presented does not meet the total understanding of what constitutes diesel fuel or jet fuel it is neither, regardless of the test results.

FOREWORD/ACKNOWLEDGMENTS

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ACRONYMS AND ABBREVIATIONS

A4A	Airlines for America (old ATA)
AFC	Aviation Fuel Committee, UK(MOD)
AFRI	African Diesel Fuel Standards
AFRL	Air Force Research Laboratories
ASTM	ASTM International
ATJ	Alcohol to Jet
ARA	African Refiners Association
BA	British Airways
B5	Diesel Fuel with 5% FAME
BOCLE	Ball On Cylinder Lubricity Evaluation
BTL	Biomass to Liquid
CARB	California Air Resources Board
CEN	European Committee for Standardisation
CH	Hydrothermal Cracking
CI	Compression Ignition
CI/LI	Corrosion Inhibitor / Lubricity Improver
CIS	Commonwealth of Independent States
CRJ	Catalysis Conversion of Alcohol to Jet
CTL	Coal to Liquid
DARPA	Defense Advanced Projects Research Agency
DLA-Energy	Defense Logistics Agency, Energy Division
DOD	Department of Defense
DSHC	Direct Fermentation to Jet
EASA	European Aviation Safety Agency
EERC-UND	Environmental Energy Research Center of the University of North Dakota
EIA	Energy Information Administration
EN	European Normung
EU	European Union
FAA	Federal Aviation Administration, US
FAME	Fatty Acid Methyl Ester, aka Biodiesel
FIA	Fluorescent Indicator Adsorption
FT SPK	Fischer Tropsch derived Synthetic Paraffinic Kerosene
GEAE	General Electric Aircraft Engines
GOST	CIS Fuel Standards
HDCJ	Hydroprocessed Depolymerized Cellulose
HEFA SPK	Hydroprocessed Esters & Fatty Acids derived Synthetic Paraffinic Kerosene
HFRR	High Frequency Reciprocating Rig, ASTM D6079
HVO	Hydrotreated Vegetable Oil
IARC	International Agency for Research on Cancer
IATA	International Air Transport Association
ICAO	International Civil Aviation Organization

ACRONYMS AND ABBREVIATIONS (Continued)

IPK	Highly branched FT SPK made by Sasol
ISLG	International Specification Liaison Group
JP-8	Jet Propellant 8
JIG	Joint Inspection Group
NATO	North Atlantic Treaty Organization
NGO	Non Governmental Organization
PQAS	Petroleum Quality Analysis System
RITA	Research and Innovative Technology Administration
SAK	Synthetic Aromatics, Kerosene boiling range
SDA	Static Dissipater Additive
SKA	Synthetic Kerosene with Aromatics
SKM	Synthetic Kerosene, Metabolically Derived
SPK	Synthetic Paraffinic Kerosene
TAN	Total Acid Number
TFLRF	TARDEC Fuels and Lubricants Research Facility (SwRI)
UK	United Kingdom
UK(MOD)	United Kingdom Ministry of Defence (UK English spelling)
ULSD	Ultra Low Sulfur Diesel
US	United States
USA	United States Army
USAF	United States Air Force
USMC	United States Marine Corps
USN	United States Navy
WHO	World Health Organization
WSD	Wear Scar Diameter

1.0 WD04 TASK 2: TRENDS IN FUEL QUALITY OF JET AND DIESEL FUELS WORLDWIDE

Fuel supplies are evolving as more highly-processed petroleum fuels, unconventional fuels, and non-petroleum fuels are increasingly making their way into the marketplace worldwide. Some of this evolution began several years ago when, for instance, environmental legislation in the U.S. mandated cleaner tailpipe emissions and as a result, the need for more highly-processed fuels, i.e., lower sulfur and lower aromatic content fuels such as California Air Resources Board (CARB) Diesel and Ultra-Low Sulfur Diesel (ULSD) fuels. The move towards developing and using non-petroleum fuels, such as biodiesel, renewable diesel/jet fuel, or Fischer-Tropsch fuels, is occurring in many countries as spurred by high volatility in the oil market, especially since 2006. In addition, much of the impetus behind transitioning to alternative fuels is tied to the desire of nations to better secure their energy supply by reducing dependence on foreign sources of oil through conversion of in-country energy resources such as tar sands, shale oil, coal, natural gas, biomass/waste streams (renewable) into transportation fuels. Furthermore, power and mobility systems are also evolving, and this may require non-traditional fuels/energy carriers as sources of energy, e.g., hydrogen for fuel cells. As these changes in the supply of fuels occurs around the world, and also in the fuels specified for future engines/equipment designs, the U.S. Military needs to understand the extent and nature of these changes and the implications regarding current and future military use. There will be some subtle and not so subtle changes in fuel compositions and associated physicochemical properties that can impact engine performance and durability, or compatibility with current (petroleum) fuels and the fuel distribution systems found in engines/vehicles such as fuel pumps, injectors, and high pressure common rail systems, or in fuel storage, distribution, or handling equipment. This project will involve assessments of the changing worldwide fuels supply with a focus on kerosene and diesel boiling range fuels, and of the impacts that varying fuel properties may have on current and future military equipment and systems.

1.1 DIESEL FUEL TRENDS

Since the early 1990's the quality of diesel fuel has been increasing around the world. This is primarily due to government mandated reductions in diesel fuel sulfur levels. At the time of this writing, while highway diesel fuel specifications in many countries set sulfur limits in the range of 10-15 ppm, maximum. In spite of this trend, there remain some countries/regions that have not mandated such strict sulfur levels. Most notable among these are selected countries in Asia and Africa.

Worldwide reductions in allowable sulfur levels have resulted in higher quality fuel for several reasons, including the following:

- The refinery processes used to reduce sulfur also tend to remove other heteroatoms (such as nitrogen) and even some aromatic compounds. In general, heteroatomic molecules and aromatic compounds tend to be the diesel fuel components most often associated with oxidation and thermal degradation of diesel fuel to form gums, varnishes, and particles.
- Removal of heteroatoms and aromatics often results in fuel with a higher cetane number, though this is not automatic.
- The emphasis on reduced sulfur has brought a greater awareness of and concentration on the overall cleanliness of fuel delivery and storage systems, which always improves the quality of the fuel delivered to the user.

In contrast, the reductions in fuel sulfur content have also resulted in diesel fuel with measurably poorer lubricity characteristics. This, in turn, has resulted in a marked increase in the use of diesel fuel lubricity additives.

In June 2012, The International Agency for Research on Cancer (IARC), which is part of the World Health Organization, released their findings regarding diesel fuel exhaust:

> *"**Lyon, France, June 12, 201,** After a week-long meeting of international experts, the International Agency for Research on Cancer (IARC), which is part of the World Health Organization (WHO), today classified diesel engine exhaust as carcinogenic to humans (Group 1), based on sufficient evidence that exposure is associated with an increased risk for lung cancer."*[1]

Such a finding, while still challenged by various stakeholders, is likely to put additional pressure on regulators to further tighten sulfur limits, especially in countries with sulfur levels above 50 ppm. This is due to the overall reduction of aromatics, and thus reducing insoluble particulates (black soot), that results from the severity of treatment needed to reach these low values.

Over the past ten years, growth in world demand for middle distillate fuels has been consistently above that for gasoline[2]. The trend is expected to continue well into the future and is manifesting itself in changes in refinery output around the world. Such changes, to both increase refinery output and modernize refinery operations, both aimed at middle distillates, should result in noticeable improvements in worldwide diesel quality. Higher quality diesel fuel should also become available in regions of the world where that has not historically been the case.

Over the past 2-4 years, both ASTM International, a nongovernmental organization (NGO), that is a source of consensus standards, and CEN (European Committee for Standardization) have been working to improve the low-temperature characteristics of biodiesel used in blends with petroleum diesel. These improvements are in the form of changes to the applicable biodiesel specifications (ASTM D6751 and European Normung (EN) 14214). For the immediate future, these changes are expected to have little effect outside the United States (US) and the European Union (EU). However, as biodiesel use in colder climates increases, these countries will look to existing specifications for guidance on low-temperature properties. Reference to the ASTM and CEN standards will ultimately result in higher quality biodiesel around the world.

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[1] International Agency for Research on Cancer, World Health Organization, Press Release No. 213, June 12, 2012.
[2] Peckham, J.; "Refining Trends;" Fuel; September 2012.

The EU is currently the leader in the mandate and/or allowance of biodiesel in their primary, highway diesel fuel specification. The EU currently allows up to 7% blends with talk of increasing to 10%. The specification, ASTM D975, allows only up to 5%. As increased usage of biodiesel becomes the norm in the EU, and the US, it is expected that usage will increase around the world. This could be a concern in colder climates or regions where less stringent biodiesel specifications are in place. This trend remains a concern for the U.S. Department of Defense (DOD) to monitor in the future.

The proposed 5th Edition of the "Worldwide Fuel Charter[3]" is out for review and comment at the time of this writing. The following quote is taken from the draft document:

> "*This proposed Fifth Edition introduces Category 5 for markets with highly advanced requirements for emission control and fuel efficiency. As many countries take steps to require vehicles and engines to meet strict fuel economy standards in addition to stringent emission standards, for diesel fuel, this category [Category 5] establishes a high quality hydrocarbon only specification that takes advantage of the characteristics of certain advanced biofuels, including hydrotreated vegetable oil (HVO) and Biomass-to-Liquid (BTL), provided all other specifications are respected and the resulting blend meets defined legislated limits.*
>
> *Other changes from the previous edition include a new test method for trace metals, an updated gasoline volatility table and updated information relating to biofuels, including ethanol, biodiesel and other alternatives to petroleum-based fuels. Category 4, as revised, will allow biodiesel in diesel fuel at levels up to five percent by volume. As countries move toward more stringent vehicle and engine requirements, fuel quality becomes more important in terms of preserving the functionality of vehicles and engines. Sulphur-free and metal-free fuels remain critical prerequisites for ultraclean and efficient emission control systems. Fuel properties play key roles in vehicle and engine emissions and performance, and the most advanced vehicles and engines require the best fuel quality – as represented in Category 5 – to meet their design potential.*"

[3] A copy of the Worldwide Fuel Charter is available from www.truckandenginemanufacturers.org

1.1.1 Standard Specifications for Diesel Fuels

Nearly every country in the world has their own national diesel fuel standard. Of course there are some regions, such as the European Union, where numerous countries use the same diesel fuel standard (EN 590 in that case). In contrast, aviation jet fuel is typically specified by one of two specifications, ASTM D1655 or United Kingdom Ministry of Defence, UK(MOD), DS9191, throughout the vast majority of the world. And some countries choose to base their specifications on ASTM D975 or EN 590. But most countries continue to maintain a national specification. Appendix A contains a compilation of diesel fuel specifications for selected countries throughout the world.

The specification for highway diesel fuel in the United States is ASTM D975, as it has been since about 1948. In recent years ASTM D975 has been revised to include an allowance for up to 5% fatty acid methyl ester (FAME), designated as B5. ASTM D975 also contains a statement that “the grades of diesel fuel oils herein specified shall be hydrocarbon oils, except as provided in 7.3 [the allowance for biodiesel], with the addition of chemicals to enhance performance, if required, conforming to the detailed requirements shown in Table 1.” The definition of hydrocarbon oil given in ASTM D975 is:

> *“hydrocarbon oil, n—homogeneous mixture or solution with elemental composition primarily of carbon and hydrogen and also containing sulfur consistent with the limits in Table 1, oxygen or nitrogen from residual impurities and contaminants and excluding added oxygenated materials.”*

At the time of this writing, there seems to be a general trend around the world that is similar to ASTM D975. There is an allowance for biodiesel in the highway diesel fuel specification, though the allowable amount may vary. And, other, non-hydrocarbon oil, blend components are not allowed in the fuel. Additionally, the source of the hydrocarbon oil (petroleum, natural gas, vegetable oil, etc.) is becoming less of a concern. The cost of these blend stocks may be a larger factor in their use, at least for the near future. Their effect on final blend properties (such as cetane number, viscosity, and low temperature operability) will also influence the extent of their use. Fuel additives can be used to mitigate some of the potentially adverse effects.

In 2012 CEN published CEN 15940, "Automotive fuels – Paraffinic diesel fuel from synthesis or hydrotreatment – Requirements and test methods." According to the specification, paraffinic diesel fuel does not meet the current requirements of European diesel fuel specification EN 590. The main differences are in distillation, density, sulfur, aromatics, and cetane. The specification also notes that the use of paraffinic diesel fuel in existing diesel engines can result in substantial reductions in regulated emissions. An effort to develop a similar specification within ASTM recently failed to pass balloting.

We are not currently aware of any widely recognized/used national or international specification for triglyceride based fuel oils (straight vegetable oil / raw vegetable oil). The same holds true for alcohol-based diesel fuels, alcohol blend diesel fuels, and water emulsion diesel fuels.

In 2006, the African Refiners Association, ARA, adopted a series of measures designed to harmonize gasoline and diesel fuel specifications, especially in Sub-Saharan Africa. The specifications, known as the AFRI standards include four grades of diesel fuel, AFRI- 1, 2, 3, and 4. The specifications have requirements for 4 key properties of diesel fuel: sulfur, density, cetane index, and lubricity. Specific requirements are given in Table 1.

Table 1. African Diesel Fuel Specifications

	AFRI-1	**AFRI-2**	**AFRI-3**	**AFRI-4**
Sulfur, mass %, max	0.8	0.35	0.05	0.005
Density @ 15°C, kg/liter, min/max	800/890	800/890	800/890	820/880
Calculated Cetane Index, min	42	45	45	45
Lubricity, HFRR @ 60°C, min	Report	Report	460	460

1.1.2 Alternative Diesel Fuel

By far, the most significant alternative diesel fuel in the international marketplace (based on market penetration) is biodiesel, FAME. Factors influencing this include:

- Length of time it has been available in the marketplace.
- Presence of standard specifications for biodiesel and biodiesel blends.
- Perceived benefit of biodiesel as a "green" fuel or renewable diesel.
- Availability of numerous sources from which to make FAME.
- Laws and regulations around the world that either encourage or mandate the use of biodiesel.
- Strong, continuing support from trade groups and government agencies to promote biodiesel usage and to improve the overall quality of biodiesel in the marketplace.

Arguably, the second most prominent alternative diesel fuel is paraffinic middle distillate fuel (PMD fuel). Generally, this fuel is characterized as paraffinic hydrocarbons in the boiling range and carbon number of commercial, middle distillate fuels (diesel and jet). It is most often made through either hydrotreating processes or Fischer-Tropsch (FT) processes. Starting materials include coal, natural gas, vegetable/plant oils, and animal fats. There is a CEN specification for this type of diesel fuel. But there is no ASTM specification for this type of diesel fuel at this time. Comparative advantages/disadvantages with biodiesel include:

- PMD fuel often has a higher cetane number.
- PMD fuel is less sensitive to oxidation and oxidative degradation.
- PMD fuel contains no oxygen so it does not have the same energy content penalty.
- PMD fuel has similar material compatibility characteristics as petroleum diesel whereas biodiesel does not.

Although PMD fuel can be made from renewable sources, it tends to be less bio-degradable than biodiesel (owing to the oxygen atoms in biodiesel). This makes PMD a potentially less-environmentally friendly fuel.

There are numerous other alternative diesel fuel sources/manufacturers, in various stages of development, in the world marketplace. The raw materials vary as do the manufacturing processes. It is likely that some of these will be successful (i.e. find a market demand) and many will not. Many of these manufacturers seem to find the jet fuel market more promising and therefore do not concentrate on diesel applications. While that may be the correct economic judgment, it should be remembered that aviation kerosene can just as easily be used as grade number 1 diesel fuel. The DOD should remain cognizant of newly emerging jet fuels that might also find their way into the diesel fuel pool.

1.1.3 Diesel Fuel Quality Data

Unfortunately, there is almost no reliable source of diesel quality information available for inclusion in this report. At the direction of TARDEC, SwRI purchased copies of diesel fuel property surveys from the Alliance of Automobile Manufacturers. The data are for both summer and winter fuels from 2009. The data were sent to TARDEC for their inspection and use but cannot be included here due to copyright restrictions. The only fuel surveys found in the open literature date back to the 1980's and 1990's. These have little relevance for today's diesel fuel. Some expectation of fuel quality in a given country/region can be gleaned from the relevant specification. Most fuel suppliers strive to meet the applicable specifications in the markets they serve. The African Refiners Association report referenced above does include some estimates of fuel quality in African countries. In general, most of the countries of interest seem to meet either AFRI-1 or AFRI-2. The data were presented in a map that cannot be reproduced in this report so the reader is encouraged to read the report for the available information.

Some additional sources of information were identified during this project. They are listed below in Table 2.

The reader is encouraged to access these sources of information for the most up-to-date information available.

Table 2. Diesel Fuel Information Resources

Name	Web Site or Mailing Address	Short Summary
U.S. Energy Information Administration	http://www.eia.doe.gov	A large volume of information on energy from many sources. Information is available for both U.S. and international.
Petroleum Quality Information System	www.dla.mil	Includes data for several types of fuel. Excellent statistical treatment.
International Association for Stability, Handling, and Use of Liquid Fuels	www.iash.net	Includes a listing of fuel specifications.
International Fuel Quality Center	www.ifqc.org	Publishes an annual summary of worldwide automotive fuel specifications. Published summary is free with a one-seat membership in the IFQC (cost is $50,000 per annum). Direct purchase is $10,000.
World Resources Institute	http://projects.wri.org/sd-pams-database	Brings together policies and measures of 18 developing countries that have.

1.2 JET FUEL TRENDS

1.2.1 Standard Specifications for Refined Jet Fuel

While diesel fuel has a wide variety of types and specifications, kerosene jet fuel is close to an ideal commodity. Essentially, there is only one kerosene jet fuel in the world. True, there is a difference in freeze point between Jet A and Jet A1 but, except for a very few flights, that is a moot point. The fact is that for all the properties that define the day to day performance of jet fuel, the requirements are uniform worldwide. ASTM D1655 Jet A is the basic, consensus standard for jet fuel. Every other kerosene jet fuel is a variation, thereof. This relationship is illustrated in Figure 1.

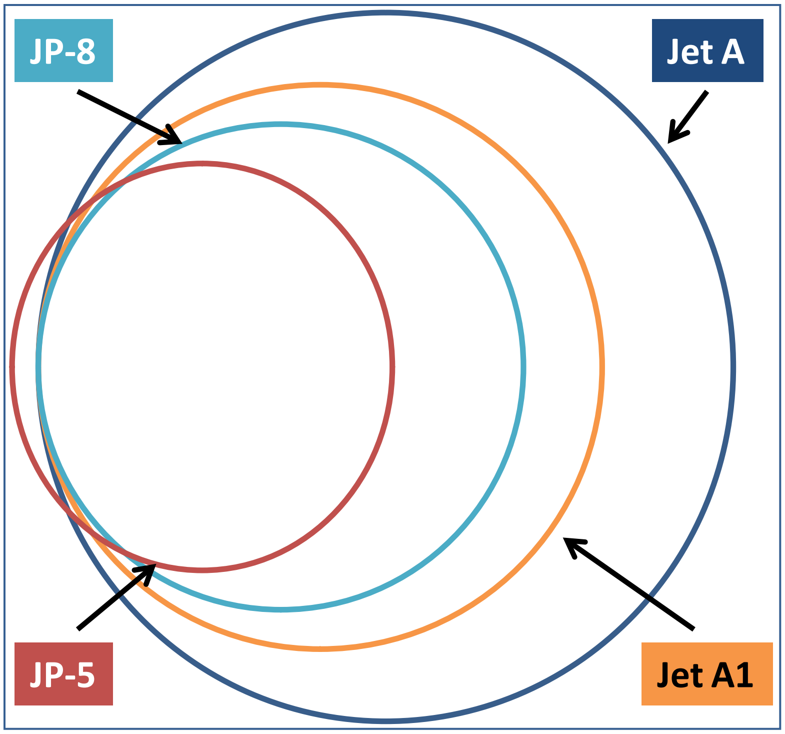

Figure 1. Venn Diagram of Kerosene Jet Fuel Types

Except for the slight bulge for JP-5 (which allows for a slightly denser fuel, 840-845 kg/m^3, than does the Jet A specification), everything meets Jet A requirement. Jet A is really the minimum acceptable jet fuel. Even an exotic fuel like JP-TS (the special high thermal stability fuel used in the U2 program) would fit in this diagram.

Why is that true? Because airplanes go everywhere and the only way the system works is if they can rely on the fuel everywhere. The world aviation community, through IATA (International Air Transport Association) with the support of the ICAO (International Civil Aviation Organization), basically dictates that if you want international service you have to provide the specified fuel. Basically, the international specification is balanced between ASTM D1655 and UK(MOD) DS91-91. How prevalent is this? Even though Russia and China have some alternative grades for military/internal use, their primary international commercial fuels are their version of ASTM D1655.

The enforcement mechanism for this requirement is the need to meet the requirements of the aircraft type certificate. All commercial aircraft are certified to use ASTM D1655 and UK(MOD) DS 91-91 fuel. These type certificate requirements are legally enforced by civil aviation authorities such as FAA (Federal Aviation Administration) and EASA (European Aviation Safety Agency). They are also policed by trade organizations such as A4A (Airlines for America), IATA and JIG (Joint Inspection Group).

The only other jet fuels of note are the partial naphtha fuels used in areas with very low surface temperatures. The primary examples are GOST TS1, used in Russia and the colder Commonwealth of Independent States (CIS) member states, Jet B, used in Alaska and Canada, and JP-4. Unlike Jet A and A1, not all aircraft are certified to use these fuels.

IATA and ASTM maintain a joint working group, the International Specification Liaison Group (ISLG), that meets twice a year to discuss specification harmonization. While the world flies on essentially a single fuel there are a variety of implementations. Some countries, like Spain and China, have their own translations of the standard methods. Other countries will only periodically update the version of the specification on which they rely, that is they might be on ASTM D1655-06 versus the current 11b or UK(MOD) DS91-91 Issue 5 versus the current Issue 7. The preference is for all countries to use the current versions but short of some radical change in the specification this is not a significant problem.

This uniformity has proved very successful as fuel is very reliable. Most of the specification debates deal with lifecycle issues, trying to increase the typical commercial service life "on the wing" beyond the typical 20,000 hrs. The only recent international incidents that might have been attributed to fuel proved to be design related (the crash of a British Airways (BA) Boeing 777 at Heathrow) and fueling operations (the hard landing of a China Southern Airbus 330 at Hong Kong) issues. The seriousness of these incidents, with the BA crash initially thought to be fueling related too, prompted IATA to ask ICAO[4] to formalize fuel specification, distribution

[4] ICAO is a UN treaty organization, the "Chicago Treaty" in this case. It sets commercial aviation regulations with which all signatories, including the United States, are obligated to abide.

and handling requirements. The new regulations, ICAO 9977 *Manual on Civil Aviation Jet Fuel Supply*, is primarily a compendium of specifications and practices that need to be followed. It codifies the use of ASTM D1655 and UK(MOD) DS91-91 as the primary kerosene jet fuel specifications.

Aviation turbine fuel is a fundamental commodity business and conformity is a clear driver. Unlike gasoline and diesel product that are split into hundreds, if not thousands, of grades, jet fuel can be thought of as single material. The specifications are recipes for crude refineries – meet these requirements and the product will be jet fuel. How big is the market? Figure 2 illustrates fuel consumption for a typical year.

Figure 2 is a combination of DLA-Energy, RITA (Research and Innovative Technology Administration) and EIA (Energy Information Administration) statistics. The world total is approximate because the latest number found was for 2007. The USAF/USA bar is for JP-8, used by the Air Force and the Army. The USN/USMC bar is for JP-5, used by Navy and Marines.

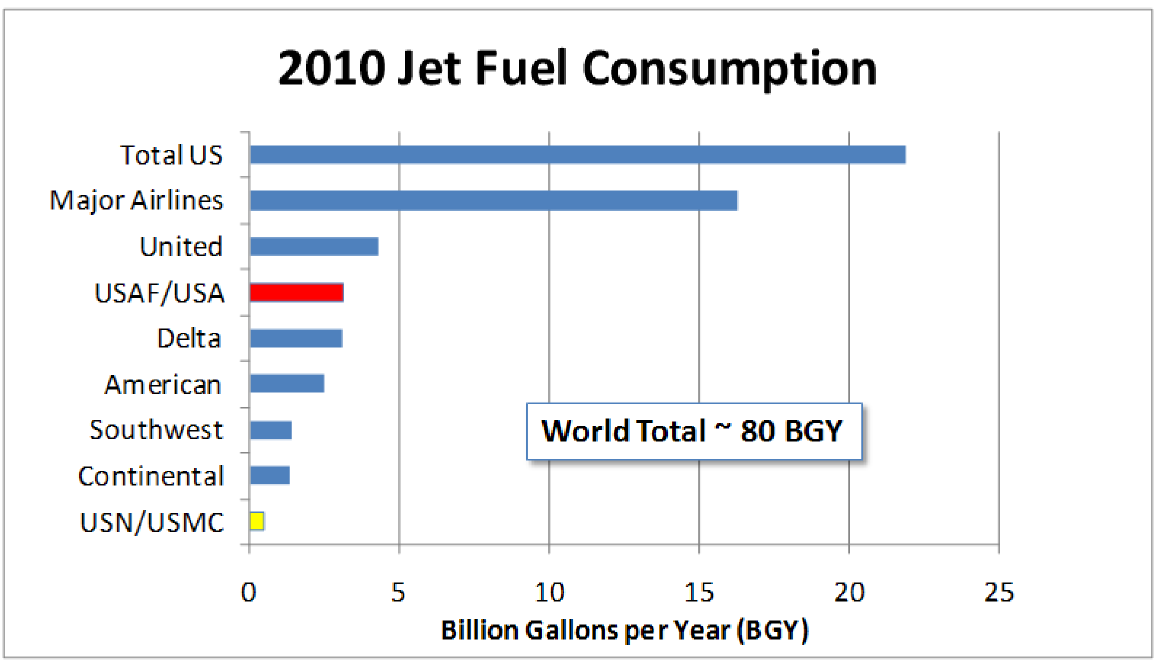

Figure 2. Jet Fuel Consumption Totals

Note that total jet fuel purchases by DLA-Energy, for all services, are ‘big airline’ large but do not dominate the business. The influence that the DOD has on commercial fuel specifications is based on the amount of research effort expended, not the purchasing power. According to 2010 DLA-Energy numbers, purchases for the Army accounted for less than 21% of total military fuel acquisition. Since several fuels are procured for Army use, the total percent of JP-8 directed to the Army would be lower still.

1.2.2 Standard Specifications for Alternative Jet Fuel

The fact that refined aviation turbine fuel is rarely an issue for commercial aviation is a testament to the five plus decades of specification activity that has resulted in excellent control. The basic concept for approving alternative fuels was defining that this experience described bounds for what is “fit for purpose” in aviation turbine engines. Thus it was reasoned that if the alternative fuel could be made to perform in the same manner it too would be fit for purpose.

Turbine fuel is used for more than power. Its heat transfer and hydraulic actuation properties are also important attributes. It also has to be compatible with the materials from which the aircraft are made and with the environments in which the aircraft is operated. Over two decades of effort, starting with the Sasol effort to supply synthetic aviation turbine fuel in South Africa, have gone into defining the key properties of aviation turbine fuel. That effort led to getting a Sasol specific approval in UK(MOD) DS91-91. The basic outline of these properties and the program conducted is found in ASTM D4054 (09 and newer), the Standard Practice for Qualification and Approval of New Aviation Turbine Fuels and Fuel Additives. This is not a rote process but an interactive journey with the aviation community in general and the airframe and power plant manufacturers in particular. The more that is known about hydrocarbons, for instance, the less exotic the testing; but each new hydrocarbon class has resulted in new evaluation recommendations.

It was not simply a matter of codifying existing practice, however. The Sasol FT SPK was approved as a sole site source in the UK(MOD) DS 91-91. An ASTM specification has to be generic in nature and offer a path to use for any appropriate source. The significant issue was that the Sasol FT process is unique in comparison to newer FT processes. The Sasol process produces

highly isomerized paraffinic kerosene where as the newer processes produce paraffin wax. That wax is put through additional processing to generate kerosene suitable for aviation use. In the study that led to the original ASTM D7566 the industry proved that both of these approaches produce kerosene suitable for aviation purposes.

When ASTM D7566 was first published in 2009 the only allowable alternative path was the use of Fischer Tropsch derived synthetic paraffinic kerosene, FT SPK. In 2011 it was modified to include the first alternative path aimed exclusively at biologically derived jet fuel components from the hydroprocessing of fats and oils, HEFA SPK. This relatively quick addition to ASTM D7566 was aided significantly by how closely the HEFA SPK resembled the kerosene generated in the Fischer Tropsch process, FT SPK. While the inclusion of a primarily renewable path was the industry goal from the outset of the standardization process, the obvious starting point was with reasonably established (nearly a decade of experience in South Africa) FT SPK.

So now there are two paths for generating alternative aviation fuel in ASTM D7566. Annex A1 allows the production of synthetic paraffinic kerosene, FT SPK, primarily from coal and natural gas but the use of biomass as a feedstock is allowed (thus providing a renewable path). Annex A2 allows the production of synthetic paraffinic kerosene, HEFA SPK, from fats and oils. Either of these SPKs may be blended up to fifty percent (depending, primarily, on density and aromatic content) with refined aviation turbine fuel. The resulting product is fit for purpose and may be used without condition, other than the standard requirements for using any refined fuel. The only interest in source would be for environmental accounting and that would only be available at the point of origin as the agreed practice is that the fuel produced by ASTM D7566 will enter commerce under either ASTM D1655 or UK(MOD) DS91-91, as allowed by both specifications.

The current version of ASTM D7566 is a milestone in the production of alternative fuels but the work is not finished. Even while the FT and HEFA SPKs were being standardized, new approaches to producing alternative aviation materials were being developed. The variety is impressive but the approaches can be narrowed to two primary topics: synthetic kerosene with aromatics (SKA) and metabolically derived kerosene (SKM). Table 3 lists the alternative processes of interest, approved and pending.

Table 3. Alternative Jet Fuel Processes

Status	Class	Process	Feedstock
Completed			
Annex A1	FT SPK	Fischer Tropsch (FT) derived SPK	Coal, Natural Gas, Biomass
Annex A2	HEFA SPK	Hydroprocessed Fats and Oils (HEFA) derived SPK	Triglyceride Oils
In the Approval Process			
	FT SKA	FT derived SKA	Coal, Natural Gas, Biomass
	ATJ SPK	Fermentation alcohol, oligmerized and hydrotreated (ATJ) derived SPK	Sugar, Alcohol
In Development			
	ATJ SAK	Catalysis to SAK, primarily aromatics	Sugar, Alcohol
	ATJ SKA	ATJ derived SKA, partial aromatics	Sugar, Alcohol
	CH SKA	Hydrothermal Cracking and Cyclization derived SKA	Triglyceride Oils
	CRJ SPK	Catalysis, oligermerized and hydrotreated derived SPK	Sugar, Alcohol
	DSHC SPK	Direct Fermentation to SPK	Sugar
	HEFA SKA	HEFA derived SKA	Triglyceride Oils
	HDCJ SKA	Hydroporcessed Depolymerized Cellulose derived SKA	Lignocellulose
SPK	Synthetic Paraffinic Kerosene		
SKA	Synthetic Kerosene with Aromatics		
SAK	Synthetic Aromatics, Kerosene boiling range		

While the general belief is that less aromatics are better (for engine life and emissions), there is a minimum requirement. This is based primarily on two needs, density and elastomer compatibility. Aircraft operation planning depends on fuel meeting a minimum density requirement. The analysis of historic fuel properties that led to setting the initial blend requirements suggested that 8.0% aromatics was an appropriate value to meet density requirements. Experience in synthetic fuel evaluations has shown that it is an appropriate level. This level is not specified for refined fuels because natural variation in the paraffin content can result in a denser product requiring less aromatic content.

In the extensive analysis of material compatibility for the proposed hydrocarbon blend materials one item has stood out as critical – proper sealing characteristics of nitrile elastomers. These materials are very common in the fleet, particularly for sealing fuel tanks. The minimum aromatic content for maintaining seal swell has not been defined but the same historical experience that pointed to 8% being a practical minimum to maintain density supports the conclusion that it is sufficient for the elastomers too.

Practical experience has shown that meeting the minimum density requirement has been a limiting factor in how much SPK can be used. Sasol, the leader in synthetic aviation turbine fuel experience, found this limitation to be a significant issue and led an effort for another single site source approval to allow synthetic kerosene with aromatics (SKA) to be approved for use in UK(MOD) DS 91-91. This is not a South African exclusive issue so the ASTM Emerging Turbine Fuel group is working toward a generic approval for SKA. Current refined fuel characteristics already limit the blending potential for SPK. On the horizon are potential limits on fuel sulfur content and, if the experience with the removal of sulfur from diesel fuel is a predictor, that could further reduce the aromatic content of refined fuel and, thus, the ability to blend in synthetic components. In the long term, producing SKA is the path to delivering a fully synthetic aviation turbine fuel.

As new processes are developed the specification issues become more complex. Every new synthetic source can built on the common experience but unique attributes will have to be addressed. Dealing with these complex issues may result in a bulky document but the aviation industry will always choose clarity over brevity. Without clarity there is too much room for interpretation.

2.0 WD04 TASK 3: FUELS FROM DEVELOPERS AND UNCONVENTIONAL SOURCES

2.1 DIESEL FUEL SAMPLES

Table 4. Diesel Fuel Samples

Sample	CL-Number	Fuel Type	Source	Type
SWD-1	CL10-0006	Fatty Nitrile	Western Biofuels (Guatemala)	Fatty Nitrile
SWD-2	CL10-0409	FT Diesel (CTL)	Sasol (South Africa)	FT Diesel (CTL)
SWD-3	CL10-0408	Green Diesel	UOP	Green Diesel
SWD-4	CL10-0407	C15 Isoprenoid	Amyris (Brazil)	C15 Isoprenoid
SWD-5	CL11-2577	FT Diesel (BTL)	Rentech	FT Diesel (BTL)
SWD-6	CL11-2921	GDiesel	Advanced Refining Concepts	GDiesel

2.1.1 Diesel Sample Discussion

The samples listed in Table 4 come from a variety of suppliers. Following is a brief description of each source and process:

1. Western Biofuels (Guatemala): A nitrile containing heterocarbon fuel being produced in Guatemala by the U.S. company Western Biofuels. They are promoting this fuel as having unique energy density based on the nitrile component.
2. Sasol (South Africa): This is fully synthetic diesel from the Sasol coal to liquid (CTL) plant in South Africa. The Sasol FT plant is unique in that it makes a full range (paraffins, cycloparaffins, and aromatics) of synthetic components. Their FT SPK (jet kerosene) is known to have a low Cetane Number but this product is not meant for use in compression ignition (CI) engines.
3. UOP (IL, USA): This fuel is from the UOP 'Green Diesel' process which essentially converts the fats and oils into paraffin waxes. The product is subsequently distilled to appropriate boiling range fractions.
4. Amyris (CA, USA): This fuel is being produced in a large scale pilot plant in Brazil by Amyris (a California biotech company). It is the first large scale use of micro-organisms to generate distillate fuel directly. The organisms convert sugars into hydrocarbons in C_5 increments. The resulting material is typically a cycloparaffinic with some aromatic content. The C_{15} product is then hydrotreated to generate a mildly branched isoparaffin with good cetane values and low temperature properties.
5. Rentech (CA, USA): This fuel is from their FT biomass to liquid (BTL) process. Unlike some other cobalt catalyzed FT processes that only make paraffins, the Rentech process also produces a small amount of aromatics. The belief is that small amount of aromatics will be sufficient to ensure elastomer compatibility.
6. Advanced Refining Concepts (NV, USA): This fuel is derived by catalytically adding methane to refined diesel fuel. Diesel fuel is worth more on a mass basis than natural gas and this concept relies on the value proposition that it is more cost effective to augment existing diesel supplies than to make diesel fuel directly from natural gas.

2.2 JET FUEL SAMPLES

Table 5. Jet Fuel Samples

Sample	CL-Number	Fuel Type	Source	Jet or Blend Stock	Type
SWJ-1	CL10-0007	Kerosine/FAME blend	GEAE (from Brazilian source)	Jet	10% FAME
SWJ-2	CL10-1346	HRJ Blend	EERC-UND	Jet	HRJ Blend
SWJ-3	CL10-1347	Isoparaffinic Kerosene	GEVO	Blend	Ferment IPK
SWJ-4	CL10-1350	Fully Synthetic Jet Fuel	SASOL (South Africa)	Jet	FSJF
SWJ-5	CL11-2578	Algal HEFA/JP-8 Blend #1	DARPA	Jet	Algal #1
SWJ-6	CL11-2579	Algal HEFA/JP-8 Blend #2	DARPA	Jet	Algal #2

2.2.1 Jet Fuel Sample Discussion

The samples listed above in Table 5 come from a variety of suppliers:

1. GEAE (General Electric Aircraft Engines from a Brazilian source): This is a sample of a research turbine fuel containing approximately 10% of $C_{8\text{-}12}$ FAME. The original testing, conducted for GEAE by TFLRF, proved that it could potentially pass for regular jet fuel.
2. EERC-UND: The Environmental Energy Research Center of the University of North Dakota participated in the DARPA program to develop processes for renewable jet fuel. This sample was prepared by blending their HEFA SPK with commercial aromatics.
3. GEVO (CO, USA): This sample is derived from their butanol to hydrocarbon conversion process. The process produces a highly isomerized product, similar to Sasol IPK. The butane building blocks result in a product with a two molecular weight distribution of mixed isomers of *iso*-dodecane and *iso*-hexadecane.
4. SASOL (South Africa): This sample is from the program that generated the only approved fully synthetic jet fuel. This particular sample failed the freeze point requirements and had to be replaced but all the other properties were identical to the material used in approval program.
5. DARPA (Defense Advanced Research Projects Agency): Following their program that led to the development of processes for HEFA SPK, DARPA started a program to develop viable processes for growing algae. Part of the deliverables for the second program was fully formulated jet fuel. These samples are from the successful programs.

3.0 WD004 TASK 4: EXPAND CETANE DATABASE

Cetane is the common name for *n*-hexadecane. It is the established high reference (100) for compression ignition (CI) qualities of distillate fuels. The comparative values are established by testing blends of cetane with the low reference (15), heptamethylnonane. For compression ignition purposes a Cetane Number values of 40 – 49 is considered adequate and values of 50 – 60 are considered premium. Values above and below these ranges have potential performance issues.

Since the US Military follows the North Atlantic Treaty Organization's (NATO) Single Fuel Forward doctrine, the CI performance of a candidate fuel is important for both diesel fuel and jet fuel. For refined fuels there are four main approaches to determining the CI performance of a distillate fuel:

1. Cetane Number by ASTM D613: This is the classic method wherein the candidate fuel combusted in a single cylinder test engine and the CI characteristics are rated ratiometrically compared to blends of the reference materials.
2. Cetane Index by ASTM D976: This value is derived from a formula that uses the density of the sample and the mid-boiling temperature (T50) from ASTM D86. This relation was calculated from data generated from a large number of refined samples.
3. Cetane Index by ASTM D4737: This value is derived from a formula that uses the density of the sample and the 10% boiling temperature (T10), mid-boiling temperature (T50) and the 90% boiling temperature (T90) from ASTM D86. This relation was calculated from data generated from a large number of refined samples.
4. Derived Cetane Number by ASTM D6890 (IQT): This value is derived by comparing the ignition delay of a sample passed into heated reservoir to the cetane engine results for a large number of refined samples.

3.1 DIESEL FUEL CI PROPERTIES

3.1.1 Test Results

Table 6. Diesel Fuel CI Properties[5]

Test	ASTM	Fatty Nitrile	FT Diesel (CTL)	Green Diesel	C15 Isoprenoid	FT Diesel (BTL)	GDiesel
CETANE Number	D613	59.3	>74.3	>74.3	60	71	45
CETANE INDEX	D976	58.9	76	77.4	71.7	73.4	46.4
CETANE INDEX	D4737	63.2	82	90.9	84.4	77.4	46.1
DCN	D6890	62.18	74.44	70.92	56.86	70.46	46.56

3.1.2 Discussion of Test Results

In Table 6 it can be seen that the three processes that generate a paraffin wax stage, the two FT Diesels and the Green Diesel, have very high cetane values. The paraffin wax is only isomerized sufficiently to get the requisite low temperature property. To the point of this comparison of methods, note the highlighted cells. These show considerable divergence between methodologies. This is to be expected as the two calculated methods, ASTM D976 and D4737, are not meant for use with synthetic fuels, which is a common problem with the casual application of specification tests to non-standard materials.

3.2 JET FUEL CI PROPERTIES

3.2.1 Test Results

Table 7. Jet Fuel CI Properties

Test	ASTM	10% FAME	HRJ Blend	Ferment IPK	FSJF	Algal #1	Algal #2
CETANE Number	D613	43.3	42.9	<19.3	39.7	47.1	45.9
CETANE INDEX	D976	39.5	48.3	53.7	46.3	51.7	43.0
CETANE INDEX	D4737	40.8	50.6	59.7	50.0	53.8	45.6
DCN	D6890	42.81	46.09	15.76	37.55	49.33	44.90

[5] For this and subsequent tables, items of particular note will be highlighted yellow. If the value listed is out of the accepted range, the text will be red.

3.2.2 Discussion of Test Results

Table 7 also shows that the Cetane Index calculations are not a reliable method for estimating the combustion characteristics of fuels from alternative sources. Note that the ferment IPK is a domestically sourced material and not the Sasol IPK that has been reported as having a very low Cetane Number. When the very low value for the Sasol material was originally discussed there was some thought that it was likely to be a single site exception to the normal synthetic kerosene material. This evaluation proves otherwise. The ferment IPK is not out of specification because there is no cetane requirement for jet fuel[6].

3.3 SUMMARY OF CI PROPERTY EVALUATIONS

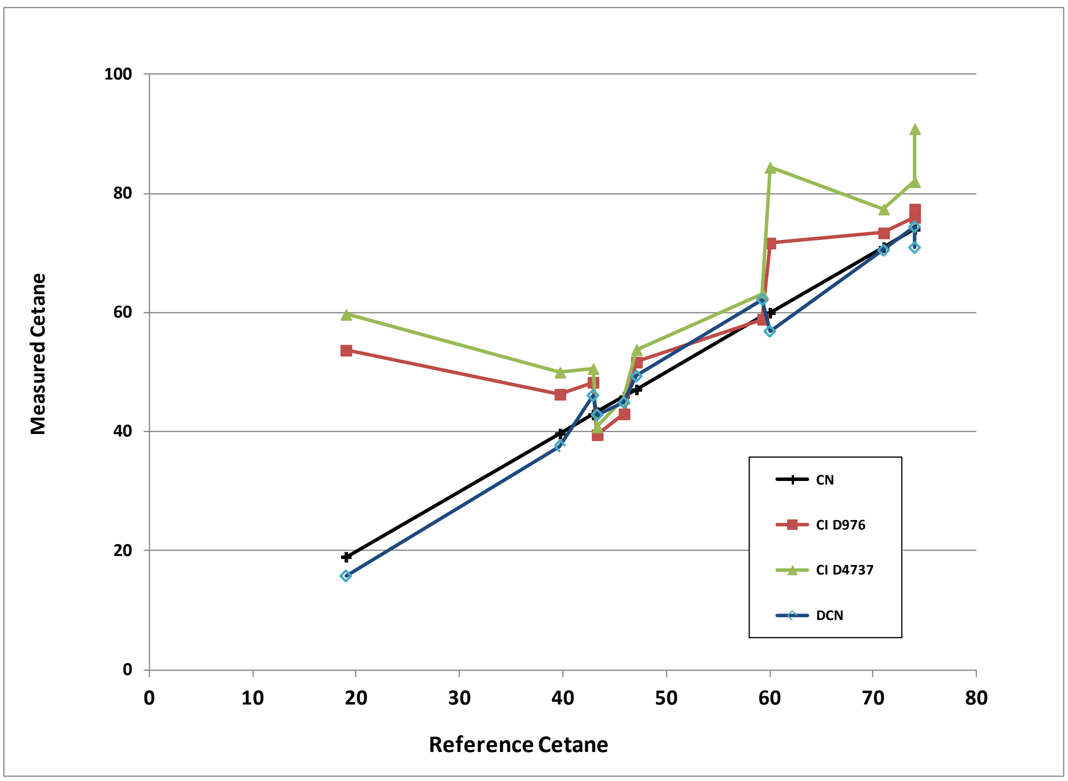

Figure 3. Comparison of Cetane Evaluation Methods

[6] Technically, the JP-8 standard requires a minimum Cetane value of 40 for synthetic kerosene used for blend stock. However, the conversion to Jet A/A1 makes that a moot point as there is no such requirement for commercial blend stocks.

In Figure 3 the comparative CI quality values are plotted for all the samples in this program. It is clear that the calculated methods have potential performance issues with alternative fuels. In the introduction to this section it was noted that the Cetane Index values were derived from physical properties of refined fuels. Those measurements do not account for fuel chemistry. Both the high and low reference fluids are C_{16} paraffins. The difference is chemical structure. The DCN test, on the other hand, responds to the same chemistry issues as does the actual cetane test engine.

4.0 WD004 TASK 5: EXPAND LUBRICITY DATABASE

4.1 DIESEL FUEL LUBRICITY PROPERTIES

All of the diesel samples were evaluated for lubricity properties by ASTM D6079, the HFRR (High Frequency Reciprocating Rig) method. They tested as received (neat) and with 200 ppm of a commercial diesel lubricity additive.

4.1.1 Test Results

Table 8. Diesel Fuel Lubricity Properties

Test	ASTM			Fatty Nitrile	FT Diesel (CTL)	Green Diesel	C15 Isoprenoid	FT Diesel (BTL)	GDiesel
HFRR	D6079								
		NEAT	WSD	354	608	585	348	360	590
		W/200ppm DLA	WSD	363	362	382	256	300	380

4.1.2 Discussion of Test Results

In Table 8 three of the neat samples exceed the ASTM D975 specification lubricity limit of 520 microns. All of the hydrocarbon samples showed a response to adding the lubricity additive. The nitrile product did not respond to the additive treatment.

4.2 JET FUEL LUBRICITY PROPERTIES

The initial plan was to test each sample by way of ASTM D5001, the BOCLE (Ball on Cylinder Lubricity Evaluator) test, as received (neat) and with two of the CI/LI (corrosion inhibitor / lubricity improver) additives approved for use with military fuels. The additives were Innospec DCI-4A and Nalco/Exxon 5403 and each was to be tested at qualified minimum effective concentration and maximum allowable concentration levels. The program was subsequently modified to include ASTM D6079, the HFRR method, testing to the same requirements.

4.2.1 Test Results

Table 9. Jet Fuel Lubricity Properties

Test	ASTM			10% FAME	HRJ Blend	Ferment IPK	FSJF	Algal #1	Algal #2
BOCLE	D5001								
		NEAT	WSD	0.52	0.79	0.63	0.76	0.62	0.59
		9 mg/l DCI 4A	WSD	0.50	0.61	0.56	0.60	0.57	0.55
		22.5 mg/l DCI 4A	WSD	0.48	0.60	0.49	0.60	0.55	0.56
		12 mg/l 5403	WSD	0.54	0.61	0.54	0.60	0.57	0.56
		22.5 mg/l 5403	WSD	0.48	0.62	0.52	0.61	0.56	0.54
HFRR	D6079								
		NEAT	WSD	410	730	670	690	720	720
		9 mg/l DCI 4A	WSD	350	700	670	710	700	680
		22.5 mg/l DCI 4A	WSD	330	710	670	700	710	700
		12 mg/l 5403	WSD	370	710	670	710	700	730
		22.5 mg/l 5403	WSD	340	690	660	710	680	730

4.2.2 Discussion of Test Results

The BOCLE results in Table 9 perform as expected for the hydrocarbon fuels. The minimum effective concentration for qualified CI/LI additives is related to the amount of additive determined to provide adequate lubricity. The fuel containing FAME has excellent lubricity to start and adding CI/LI provides little benefit.

The HFRR results in Table 9 for the hydrocarbons are all failures, if one were evaluating the material as #1 Diesel. HFRR basically responds to heavy doses of additive, well above the range typically used for CI/LI. The FAME used in the first sample is similar in structure to commercial diesel lubricity additives so it is not surprising that it would pass the HFRR test. What is interesting is the appearance of a synergistic effect of adding a fuel parts per million of the standard CI/LI additives.

4.3 SUMMARY OF LUBRICITY PROPERTY EVALUATIONS

The data generated in Table 8 offer some assurance that as alternative materials move into the diesel fuel market the lubricity additives will provide adequate performance. That is only effective, however, in countries/regions that require diesel fuel meet minimum lubricity standards.

The primary tactical fuel for the U.S. Army is jet fuel with additives, so it may be a concern that Table 9 shows the jet fuels failing the diesel fuel lubricity test. This issue is well known but the experience at TFLRF (which has been working on the use of jet fuel in diesel engines since the single fuel forward doctrine was initiated) is that the approved additives in jet fuel give adequate lubricity performance in CI engine systems.

5.0 WD004 TASK 6: INSPECTION PROPERTIES FOR EMERGING FUELS

5.1 DIESEL FUEL INSPECTION PROPERTIES

All of the diesel samples were tested in accordance with ASTM D975, the Standard Specification for Diesel Fuel Oils. (Please refer to this document for information about specific limits.) Since the waivers for sulfur content only apply to the use of jet fuel in U.S. Army vehicles the results are evaluated in respect to the requirements for #2-D S15 grade diesel (ULSD).

5.1.1 Test Results

Table 10. Diesel Fuel Inspection Properties

Test	ASTM		Fatty Nitrile	FT Diesel (CTL)	Green Diesel	C15 Isoprenoid	FT Diesel (BTL)	GDiesel
Flash Point	D93	°C	67.5	59.5	81.0	104.0	52.0	55.5
Water and Sediment, % Vc	D2709	Sediment	0.01	0.01	0.01	0.01	0.005	0.001
		%/°C						
Distillation	D86	IBP	153.8	168.1	192.8	228.4	156	173.6
		5%	226.5	200.2	215.8	244.3	175.3	192.3
		10%	243.7	207.9	225.4	244.7	179.8	198.1
		15%	251.5	214.8	234.3	244.8	187.6	204.3
		20%	258.1	221.2	246.7	244.8	195.1	209.6
		30%	268.4	234.5	263.3	244.9	213.2	222.4
		40%	276.8	249.7	274.8	244.5	233.5	235.9
		50%	284	265.9	281.7	245	253.3	248.4
		60%	291.9	283.2	285.7	245	272.4	261.9
		70%	303	300.8	288.8	244.7	289.9	275.1
		80%	316.3	320.4	291.5	245.2	306.8	288.6
		90%	331.3	343.5	294.9	245.5	323.8	306.8
		95%	345	358.3	299.1	246	333.8	324.5
		FBP	349.1	361	307.2	247.8	339	344
		Residue, %	1.8	1.9	1.3	1.7	1.5	1.4
		Loss, %	1.8	0.6	1.1	0.4	0.4	0.2
Viscosity 40 °C	D445	mm^2/s	3.41	2.58	2.66	2.34	2.04	2.22
Ash	D482	mg/l	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Sulfur	D5453	mg/kg	29.7	0.6	0.8	1	0.5	5.2
Copper Corrosion	D130	CuCorr	1B	1A	1A	1A	1A	1A
Cetane number	D613	CN	59.3	>74.3	>74.3	60	71	45
Cetane Index	D976	CI	58.9	76	77.4	71.7	73.4	46.4
Aromatics	D1319	Aromatics, vol %	73.3	0.7	0.9	0.8	1.7	22.7
		Olefins, vol %	9.2	0.8	0.6	0.5	0.5	2
		Saturates, vol %	17.5	98.5	98.5	98.7	97.8	75.2
Cloud Point	D2500	°C	8	0	-3	<-54	-10	-15
CFPP	D6371	°C	1	-5	-9	-35	-14	-23
Ramsbottom Carbon Residue on 10% dist. Residue	D524	Mass %	0.10	0.02	0.03	0.03	0.05	0.05
HFRR @ 60°C	D6079	WSD, mm	0.354	0.608	0.585	0.348	0.360	0.590
Conductiviy pS/m	D2624	pS/m	1000	0	2	1	54	0

5.1.2 Discussion of Test Results

The most notable thing about the diesel samples, as received, is how bad the fatty nitrile sample from Western Biofuels smells. Other than that, all of the samples appear to be normal diesel fuel type products. Still there are highlights from the analyses conducted for the inspection analyses listed in Table 10 that need some discussion:

1. Flash Point **–** The Amyris C_{15} Isoprenoid diesel has a very high flash point, 104°C. While there is no upper limit in the specification for diesel fuel there are concerns among gas turbine OEMs (original equipment manufacturers) about this. There is a General Electric specification for industrial gas turbines that limits maximum flash point for distillate fuels to 93°C.
2. Distillation **–** The Amyris C_{15} Isoprenoid fails the required minimum T90 of 282°C. However, that requirement is waived for low temperature diesel and, with a Cloud Point of <-54°C, this fuel clearly meets that criteria. Another issue with that fuel is the flat distillation curve. The other samples have relatively normal curves.
3. Sulfur **–** The fatty nitrile fuel exceeds the allowable sulfur limits for ULSD. All the other processes are essentially sulfur free.
4. Aromatics **–** The fatty nitrile material has a very high reported aromatic content, at 73.3%. This is not a specific issue since the Cetane Number is >40 but it seems unusual. Since the producer says this is fatty nitrile, RCO-CN, and not aromatic nitrile, Ar-CN, this result may be related to an interference with the test dyes, thus effecting the performance of the FIA test. (ASTM D1319).
5. Conductivity **–** The fatty nitrile once again has the odd result. At 1,000 pS/m it suggests the material has natural conductivity. The other three samples are out of specification but that is just from the lack of adding static dissipater additive (SDA).

5.2 JET FUEL INSPECTION PROPERTIES

All of the jet fuel samples were tested in accordance with the requirements of MIL-DTL-83133G[7], the Detailed Specification for Turbine Fuel, Aviation, Kerosene Type. (Please refer to this document for information about specific limits). As noted in Table 5 all of the samples, except for the Ferment IPK from GEVO, are fully formulated jet fuels so they would be expected to meet the specification requirements.

5.2.1 Test Results

Table 11. Jet Fuel Inspection Properties

Test	ASTM		10% FAME	HRJ Blend	Ferment IPK	FSJF	Algal #1	Algal #2
Saybolt	D156	Rating	15	21	30	15	16	1
TAN	D3242	mgKOH/kg	0.019	0.002	0.017	0.003	0.004	0.034
Aromatics	D1319	Aromatics, vol %	18.4	23.6	0.9	10.1	19.9	17.1
		Olefins, vol %	1.2	0.4	0.6	2.9	1	1
		Saturates, vol%	80.4	76	98.5	87	79	81.9
Sulfur	D4294	Mass %	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mercaptan	D3227	Mass %	<.0003	<.0003	<.0003	<.0003	0.0007	0.0006
		%/°C						
Distillation	D86	IBP	159.9	150.6	171.3	169.3	153.7	168.9
		5%	171.6	169.7	177.0	175.5	166.5	177.0
		10%	171.9	174.3	177.9	176.6	169.2	178.2
		15%	179.1	179.0	178.6	177.6	173.7	180.0
		20%	182.2	183.2	179.0	178.4	176.7	182.1
		30%	189.8	190.0	180.2	181.2	184.1	186.1
		40%	197.6	195.9	181.7	183.9	193.0	190.5
		50%	206.2	202.0	182.1	187.5	205.2	195.5
		60%	215.9	208.6	183.4	191.6	222.0	202.8
		70%	226.1	218.1	187.3	197.5	238.9	211.4
		80%	238.9	233.5	192.9	205.6	252.1	227.0
		90%	254.4	256.9	218.6	220.0	262.9	238.2
		95%	268.6	269.8	240.2	243.7	269.3	249.2
		FBP	279.3	275.2	248.3	264.1	273.8	259.9
		Residue, vol %	1.5	1.4	1.5	1.8	1.3	1.2
		Loss, vol %	1.1	1.2	1.3	1.2	0.7	0.4
		T50-T10	34.3	27.7	4.2	10.9	36.0	17.3
		T90-T10	82.5	82.6	40.7	43.4	93.7	60.0

[7] The current version is issue H Amendment 1 but the issue G was in place when this testing was conducted.

Table 11. Jet Fuel Inspection Properties (Continued)

Test	ASTM		10% FAME	HRJ Blend	Ferment IPK	FSJF	Algal #1	Algal #2
Flash	D93	°C	52.5	46.0	48.0	52.0	45.5	54.0
Density	D4052	kg/m^3	819.4	792.8	759.7	781.9	787.7	799.2
Freezing Point	D5972	°C	-47.2	-63.8	<-81	-23.6	-37	-66
Viscosity @ -20°C	D445 LT	mm^2/s	5.2	4.1	4.88	4.18	4.32	3.97
Heat of Combustion, net	D4809n	MJ/kg	42.1	43.2	43.9	43.6	43.5	43.4
	D3338		43.1	43.3	44.0	43.6	43.4	43.3
Hydrogen Content	D3701	Mass %	13.45	14.04	15.54	14.63	13.53	13.89
Smoke Point	D1322	mm	17.0	26.5	28	30	25	26
Napthalene	D1840	vol %	1.46	1.43	0.14	0.14	0.02	0.13
Calculated Cetane	D976	CI	39.5	48.3	53.7	46.3	51.7	43
Copper Strip	D130	Rating	1A	1A	1A	1A	1A	1B
JFTOT @ 275°C	D3241	°C	275	275	275	275	275	275
		VTR	1	<2	<2	<2	2	2
		DP, mmHg	0	0	0	0	1	0
Existent Gum	D381	mg/dl	0.7	0.8	<0.5mg	2.7	0.9	11.2
MSEP	D3948	Rating	91	95	98	82	92	86
FSII	D5006	vol %	0.00	0.00	0.00	0.00	0.11	0.11
Conductivity	D2624	pS/m	210	366	363	370	129	131

5.2.2 Discussion of Test Results

Unlike the Diesel Fuel samples, there were no unusual samples among the Jet Fuels. There were however several issues from the testing that bear discussion:

1. Saybolt Color – While this is a 'Report' item, it is worth noting the very low value for the second DARPA sample.
2. Total Acid Number (TAN) – Three samples exceed the allowable value for JP-8 (0.015) but all are well within the limit for Jet A (0.10).

3. Aromatics – The Ferment IPK actually exceeds the limits for aromatics generally accepted for a synthetic paraffinic kerosene. However, FIA is not scoped for such a low number and running the more appropriate ASTM D2425 GC-MS test was not part of this program. More interesting is the high level of aromatics in the blended samples. While the EERC and DARPA samples are based on HEFA SPK technology they are blended with commercial aromatics and thus have much higher aromatic contents than was the case with the blended fuels in the program that resulted in the approval of HEFA SPK.

4. Distillation – All of the samples meet the distillation requirements as currently written. The very narrow T50-T10 of the GEVO IPK may be a problem however. The similar Sasol IPK has a wider T50-T10 but a narrower T90-T10. The GEVO data reflects the fact that it is specified to be 90-95% C_{12} IPK and 5-10% C_{16} IPK. The Sasol FSJF (50% Sasol IPK), as seen below, was considered to have a barely adequate distillation curve based on work at Rolls Royce. A fuel with 50% GEVO IPK would require specific testing against the same criteria.

5. Freezing Point – Two samples, Sasol FSJF and DARPA Algal #1, failed to meet the freeze point requirements for either JP-8 or Jet A. Since the Sasol FSJF sample had failed in previous testing it is good that the inspection methods show consistency.

6. Heat of Combustion – In a previous round of testing, conducted for GEAE, the Heat of Combustion by bomb calorimetry, the referee method, was the only thing the 10% FAME fuel failed. It passes using the calculated method, ASTM D3338. Hydrogen content by the specified method also meets the requirement. In the earlier testing, ASTM D5291 Carbon Hydrogen analyses showed that while the hydrogen content was the same it was missing about 2% of the requisite carbon.

7. Smoke Point – In this testing the Smoke Point of the FAME containing fuel fails. That value, however, is well within the Reproducibility of the method and could easily pass. As received, the sample had a Smoke Point value of 24. This suggests some form of degradation but other than a slight change in color, from 17 to 15, a slight increase in residue from 0.0 to 0.7 mg/dl, there is no other evidence of a significant change. The ASTM D3241 value still passes at 275° which is reasonably consistent with an original Breakpoint value of 290°C.

8. Copper Strip Corrosion – Modern jet fuels are so routinely rated 1A in this test that a still passing rating of 1B is noteworthy.

9. Existent Gum – The second DARPA sample, Algal #2, fails the specification requirements, < 7mg/dl, for existent gum. However, it is within the limits, <14 mg/ dl per MIL-STD-3004, for intra-governmental transfers.

5.3 SUMMARY OF INSPECTION PROPERTY EVALUATIONS

Perhaps the most important take away from this testing is how well the two non hydrocarbon fuels, the nitrile diesel and the FAME containing jet, came so close to meeting the right specification requirements. The specifications are written for fuel refined from petroleum. The standard test methods are the tests needed to confirm that the desired product has been derived from same. Meeting those requirements, alone with a material that is neither refined nor potentially not a hydrocarbon, does not make the material fit for purpose.

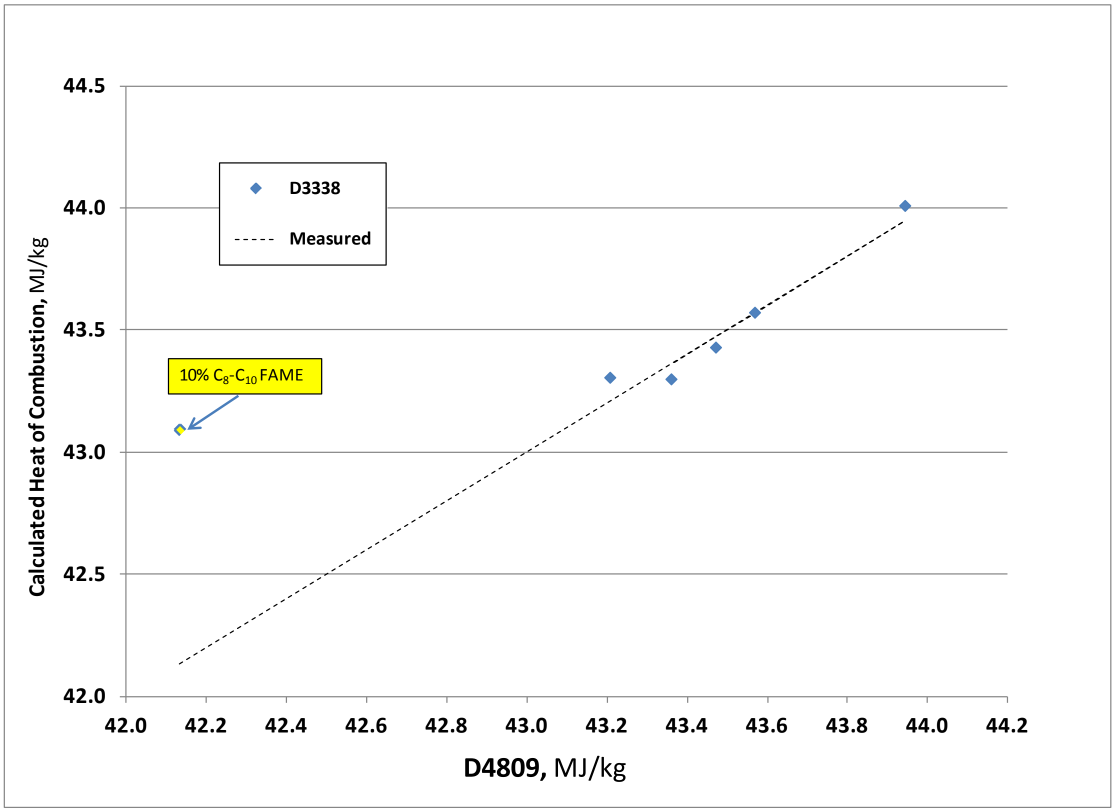

Figure 4. Potential Error from Estimation of Heat of Combustion

As an example of the problem, consider the data presented in Figure 4 wherein the calculated Heat of Combustion, by ASTM D3338, is compared to the referee value from ASTM D4809. Like the discussion on calculated Cetane Index in Section 3.0, the calculated heat of combustion is derived from, primarily the physical properties of the material in question. There is no way to know that the needed carbon is missing.

Diesel Fuel and Jet Fuel both have many required properties that are not tested with every batch. The purpose of a specification table is not to define every aspect of a fuel but to control those properties which are not a natural product of the process involved. The key task for dealing with non-refined feedstocks has been defining those properties which, when applied as a specification, will assure the resulting product is fit for purpose. This understanding only applies for the process for which the alternative specifications, such as Annexes in ASTM D7566 or the ASTM D6751 specification for B100 FAME, are developed. Comparing test results to specifications for approved processes is a good way to evaluate potential processes but it is not proof of equivalence.

This specification requirement versus fit for purpose necessities presents a hazard when evaluating unknown samples. The testing is premised on the idea that the sample presented is the material it purports to be. For a research lab, such as Air Force Research Laboratories (AFRL), the Naval Research Laboratories (NRL) or TFLRF, finding the truth of the matter is relatively simple, should the need be apparent. Most of the labs in the fuel transportation and delivery system would not be able to readily identify these issues. For a system such as PQAS (Petroleum Quality Analysis System), the mobile lab developed by the U.S. Army, whose job includes evaluating captured samples, perhaps additional test capability need to be deployed to spot fuel issues such as the ones discussed in this report. One potential approach would be to use mid-infrared (IR) technology. It definitely would be able to see distinct heterocarbon absorption peak, and there is a possibility it could be used to estimate the ratio of iso and normal paraffins. Certainly, some form of heterocarbon analysis should be a routine practice for any material of unknown source being offered for evaluation. The standard specification values are for materials from the expected source (as described in the document) and not for any material that tests to the same values.

The key point to understand about specification testing is that it is only as reliable as the quality and integrity of the sample. If the material presented does not meet the total understanding of what constitutes diesel fuel or jet fuel, it is neither, regardless of the test results.

6.0 CONCLUSIONS

6.1 DIESEL FUEL

- There is a worldwide trend to reduce the amount of sulfur in diesel. Reduction of total sulfur in diesel fuel will generally result in a cleaner-burning fuel with improved stability characteristics.
- Refining processes to reduce total diesel fuel sulfur often result in fuel with inadequate lubricity characteristics. This, in turn, necessitates the addition of lubricity improving additives.
- Worldwide trends to increase the use of diesel fuel from alternative sources have introduced uncertainty into the diesel fuel market. This is because diesel fuel from non-traditional sources may not perform as needed or expected in the engine.
- Storage and handling problems may also be caused by the use of non-traditional diesel fuel or fuel blend stocks.
- The authors were unable to locate any readily available, reliable database of worldwide diesel fuel properties. National specifications are available but these may not truly reflect the quality of fuel in the country. Some data are available through subscription services but often are limited in regions of the world they cover.
- Data already collected by the Department of Defense, in PQIS, may be the best source available at this time.

6.2 JET FUEL

- While diesel fuel has a wide variety of types and specifications, kerosene jet fuel is close to an ideal commodity. Essentially, there is only one kerosene jet fuel in the world.
- Fuel specification uniformity is moving from an informal status, driven by the need to conform to type certificate requirements, to a formal status, dictated by ICAO regulations.
- The influence that the DOD has on commercial fuel specifications is based on the amount of research effort expended not the purchasing power. DLA-Energy Jet Fuel Purchases are in the 'major airline' class.
- The development of alternative jet fuel is driven by the need to maintain conformity with the existing experience. Alternatives may only be unique in source, not in application.
- There is a general consensus that following the principles of ASTM D4054 and developing a process specific approval is the appropriate method for introducing alternative turbine fuel source.

6.2.1 Alternative Fuel Samples

- While there is a lot of interest in providing alternative fuels there is a lot less actual product being generated.
- For diesel fuel, the primary alternative fuel source is FAME. This is driven by regulatory and commercial priorities.
- There is some interest in the use of synthetic paraffinic middle distillate in diesel due to its superior cetane properties and generally cleaner combustion properties.
- There are more unique approaches being developed for jet fuel than for diesel fuel because that is where the government support money is being focused.
- Six unique diesel samples and six unique jet fuel samples were obtained for the program. One sample in each group had a heterocarbon compound not typically associated with the category and these proved challenging to the reliance on specification testing.

6.2.2 Cetane Properties

- Since the U.S. Military follows the North Atlantic Treaty Organization's (NATO) Single Fuel Forward doctrine, the CI performance of a candidate fuel is important for both diesel fuel and jet fuel.
- Alternative fuel components can be out of the expected range of cetane values.
- Cetane Index calculations are not reliable methods for estimating the combustion characteristics of fuels from alternative sources. They do not account for chemistry related combustion effects.
- DCN responds to the same chemistry issues as does the actual cetane test engine.

6.2.3 Lubricity Properties

- All of the hydrocarbon diesel samples responded to the addition of lubricity additive in a normal fashion.
- All of the hydrocarbon jet fuel samples responded to the addition of CI/LI additives in a normal fashion.
- The heterocarbon containing samples, the nitrile diesel fuel and the 10% FAME jet fuel, had excellent lubricity as measured by the method. Use of either, in practice, would necessitate rig tests to confirm the standard methods are sufficient.
- The HFRR method cannot be used to evaluate the lubricity of a hydrocarbon jet fuel using the approved CI/LI additives, at specified treatment rates.

6.2.4 Inspection Properties

- There were some failures, as might be expected, but it was more remarkable how often something that clearly is not suitable as diesel or jet fuel passed the conformance tests.
- Standard specification testing presents a hazard when evaluating unknown samples. The testing is premised on the idea that the sample presented is the material it purports to be.

- If the material presented does not meet the total understanding of what constitutes diesel fuel or jet fuel it is neither, regardless of the test results. In fuel specifications, the requirements (typically Table 1) do not cover every required physical and chemical property of the fuel, just those tests needed to assure the correct product has been made from the anticipated source. For instance, refining diesel or jet fuel from petroleum.
- The existing fuel acquisition, transportation and delivery quality assurance system would not be able to readily identify non fuels. In areas without sufficient quality infrastructure, this could be guarded against with instruments, such as infrared spectrophotometers, that could readily detect significant (% level) heterocarbon levels.

7.0 RECOMMENDATIONS

- As diesel fuel quality and composition continues to change, the Army should continue their efforts to remain cognizant to the changes.
- As commercial jet fuel specifications continue to consolidate, the Army should continue to participate in their maintenance and development. While this might not result in a fuel better suited for the entire mission it may well prevent it from becoming less suitable.
- The Army should continue to conduct research on turbine fuel properties and encourage a continued effort across the services. The research efforts funded by DOD are the primary lever for assuring that jet fuel continues to meet mission needs.
- The Army needs to conduct research for a new way to estimate compression ignition quality. Mid-IR, for instance, can see the ratio of CH_2 to CH_3 which might be used to augment the physical properties currently used to determine Cetane Index.
- The Army should look to Mid-IR techniques to identify heterocarbon fuels, such the FAME containing and nitrile materials evaluated in this program.

APPENDIX A – SELECTED DIESEL FUEL SPECIFICATIONS

Table A–1. Diesel Fuel Specifications

	United States		European Union	Afghanistan	Angola	Australia	Brazil		China		India		Japan	Kenya	Libya		Nigeria
National Diesel Fuel Specification	ASTM D975		EN 590			Fuel Quality Standards Act 2000	Portaria ANP N° 310		GB/T 19147	DB11/239	IS 1460:2005 / Bharat II	IS 1460:2005 / Bharat III	JIS K 2204	KS 1309 -1:2003			
Grade Number or Name	No. 1	No. 2			Gasoleo		Interior-"B"	Metropolitan-"D"	(3) [Voluntary]	(4) [Beijing]	HSD Nationwide	HSD Metro		Automotive Gasoil	No. 1	No. 2	Automotive Gasoil
Sources of Relevant Specification Information and/or to Purchase Specification	www.ASTM.org		www.cen.eu		IFQC	IFQC	IFQC	IFQC	IFQC	IFQC	IFQC	IFQC	IFQC	IFQC	IFQC		IFQC
Ash, wt%, max	0.01	0.01	0.01		0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	---	0.01	0.01	0.01	0.01
Cloud Point, C, max	Regional & Seasonal	Regional & Seasonal	Regional		---	---	---	---	---	---	---	---	---	Report	4(s) -1(w)	4(s) 4(w)	4
Cetane Number, min	40	40	51		---	---	42	42	45	47	48	51	45	---	---	---	---
Cetane Index, min	40	40	46		50 / 45	46	---	---	43	46	46	46	45	48	52	45	---
Density @ 15C, kg/m3, min -- max	xxx	xxx	820-845		820 min	820 - 850	820 - 880 (20°C)	820 - 865 (20°C)	800 - 840 (20°C)	800 - 840 (20°C)	820 - 860	820 - 845	---	820 - 870 (20°C)	Report	Report	820 min
Distillation																	
T50					Report	---	245 - 310	245 - 310	300 max	300 max	---	---	---	---	---	---	---
T85			350 max		---	---	370 max	360 max	---	---	350 max	---	---	---	350 max	350 max	---
T90	288 max	282 - 338			385 max / 365-370	---	---	---	355 max	355 max	---	---	360 max	365 max	---	---	357 max
T95			360 max		---	360	---	---	365	365	370	360	---	---	---	---	---
FBP					Report	---	---	---	---	---	---	---	---	---	---	---	385
Flash Point, C, min	52	38	55		66	61.5	38	38	45	55	35	35	45	60	60	60	65
Lubricity @ 60 C, awsd, microns, max	520	520	460		---	460	---	460	460	460	460	460	---	---	---	---	---
Total Sulfur, ppm, max	15	15	10		3,000	50	2,000	500	500	50	500	350	10	5,000	5,000	5,000	3,000
Viscosity @ 40°C, mm2/s	1.3 - 2.4	1.9 - 4.1	2.0 - 4.5		2.2 - 5.8 / 2.2 - 5.5	2 - 4.5	2 - 5 (37.8°C)	2 - 5 (37.8°C)	1.8 - 7 (20°C)	1.8 - 7	2 - 5	2 - 4.5	1.7 min	1.6 - 5.5	2.0 - 5.0	3.2 - 5.8	1.6 - 5.5

Table A-1 (Cont'd). Selected Diesel Fuel Specifications

	Pakistan	Peru		Russia				Saudi Arabia		South Africa	Turkey	Turkmenistan	Venezuela
National Diesel Fuel Specification	PSI	NTP 321.003		GOST 305 -82		GOST R 52368 - 2005		A-873	A-868		EN 590	GOST 05766698-06-2005	COVENIN 662:1998
Grade Number or Name	HSD	No. 1	No. 2 (D2 S-350 / S-50)	Type 1 / Type 2	Premium	Type 1 / Type 2 Sulfur Free -- Temperate	Type 1 / Type 2 Sulfur Free -- Winter and Arctic	Diesel -- National	Low-Sulfur Diesel -- City	Automotive Diesel	Low Sulfur Diesel / Sulfur-Free Diesel		
Sources of Relevant Specification Information and/or to Purchase Specification	IFQC	IFQC						IFQC		IFQC	IFQC	IFQC	IFQC
Ash, wt%, max	0.01	0.01	0.01	0.1	0.008	0.01	0.01	0.01	0.01	0.01	0.01	---	0.01
Cloud Point, C, max	9(s) 6(w)	---	---	-5(s) -25(w)	-5(s) -25(w)	---	-10/-16/-22/-28/-34	12(s) 6(i) 2 (w)	---	-4(w) 3(s)	---	---	---
Cetane Number, min	---	40	51	45	45	51	49 - 47	---	---	---	51	---	43
Cetane Index, min	45	40	46	---	---	46	46 - 43	45	45	45	46	45	---
Density @ 15C, kg/m3, min -- max	Report	---	820 - 845	860(s) 840(w) 830(a) max 20°C	860(s) 840(w) 830(a) max 20°C	820 - 845	800 - 845	Report	Report	820 - 875	820-845	---	---
Distillation													
T50	Report	---	---	280(s) 280(w) 255(a) max	280(s) 280(w) 255(a) max	Report	Report	---	---	---	Report	---	---
T85	---	---	---	---	---	350 min	350 min	350 max	350 max	---	---	---	---
T90	365 max	288 max	---	---	---	Report	Report	---	---	362 max	Report	---	360 max
T95	---	---	360	---	---	360 max	340 - 360	---	---	---	360	---	---
FBP	---	---	---	---	---	---	---	Report	Report	---	---	---	---
Flash Point, C, min	54	38	55	40(s) 35(w) 30(a)	40(s) 35(w) 30(a)	55	55	55	55	55	55	---	60
Lubricity @ 60 C, awsd, microns, max	---	---	460	---	---	460	460	---	---	460	460	---	---
Total Sulfur, ppm, max	10,000	1,500	350 / 50	2,000 / 5,000 (s)(w) 4,000 (a)	2,000	350 / 50 / 10	350 / 50 / 10	5,000	800	500	10	---	5,000
Viscosity @ 40°C, mm2/s	1.5 - 6.5	1.3 - 2.4	2 - 4.5	3-6(s) 1.8-5(w) 1.5-4(a) (20°C)	3-6(s) 1.8-5(w) 1.5-4(a) (20°C)	2 - 4.5	1.5 - 4	1.9 - 4.1	1.9 - 4.1	2.2 - 5.3	2 - 4.5	---	1.6 - 5.2